Optimization of Esterification and Transesterification of High FFA Jatropha Curcas Oil Using Response Surface Methodology

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***Abstract*- Optimization of parameters for reduction of Free Fatty Acid content (FFA) of *Jatropha curcas* oil (JCO) and maximization of yield of *Jatropha curcas* biodiesel (JCB) (%) has gained importance for boosting the fuel industry and improving the production efficiency of biodiesel. A five-level-four-factorial Central Composite Design (CCD) using Response Surface Methodology (RSM) was employed to optimize the process variables for minimizing the FFA of JCO and maximizing the JCB (%) yield. The high FFA (14.6%) of JCO could be reduced to 0.34% by its pretreatment with methanol (6.5:1) using H_2SO_4 as catalyst (1.5% v/v) in 125 min time at 50^{o}C temperature. A JCB yield of 98.3% was achieved with methanol/oil molar ratio (11:1) using NaOH as catalyst (1% w/w) in 110 min time at 55^{o}C temperature. Second-order model equations were obtained to predict the FFA content and JCB yield as a function of input parameters. The models can be successfully adopted in fuel industry to reduce the FFA content of JCO and maximize the yield of methyl esters, thereby, improving the economy of the process. The prepared JCB conformed to the ASTM and IS standards specifications.**

Keywords*- *Esterification; Transesterification; Optimization; JCB Yield; FFA Content

I. INTRODUCTION

The growing environmental concerns due to increasing carbon dioxide emissions, global warming, declining petroleum reserves and rising crude oil prices have resulted in worldwide attention to biodiesel. With growing human population, more land is needed to produce food for human consumption, which poses a potential challenge to biodiesel production. *Jatropha curcas* oil (JCO) is a plant based feedstock that is unsuitable for human consumption and could be the best feedstock for biodiesel production [1]. However, the properties of these oils are not suitable to be used in engines. They have high viscosity, high flash point and low calorific value than diesel fuel, thus, making them unsuitable to be used in diesel engines. This necessitates the need to go for modification in the oils to make their properties suitable for engine use. Transesterification is the most suitable method to go for modification in oils. The type of transesterification is chosen based on the Free Fatty Acid (FFA) content of the oil. For high FFA oils, two step acid-base catalyzed transesterification process is adopted. The FFA of the oil is first reduced to less than 1% in acid catalyzed esterification process. The oil with reduced FFA is further subjected to base catalyzed transesterification process for production of biodiesel [2]. This process involves many parameters that affect the reaction and optimizing so many reaction factors require large number of experiments, which is laborious, time consuming, and economically non-viable. Response surface methodology (RSM) is a useful statistical technique for the evaluation or optimization of complex processes, as it reduces the number of experiments required to achieve ample data for a statistically pertinent result. Tiwari *et al*. [3] have optimized three reaction variables viz. catalyst concentration, reaction time and methanol quantity using five-level-three-factor Central Composite Rotatable Design (CCRD) based on RSM for the reduction of high FFA in JCO to <1% in 34 experiments. Two variables viz. methanol quantity and reaction time were optimized in 21 experiments to maximize the *Jatropha curcas* biodiesel (JCB) yield to 99%. Similarly, Boonmee *et al*. [4] have studied the effect of three process variables viz. methanol/oil molar ratio, catalyst concentration and reaction time on the methyl esters yield of JCO using a central composite design (CCD) of 20 experiments and achieved 99.87% biodiesel yield. Other groups have applied RSM to optimize process factors for biodiesel production, using rapeseed oil, soybean oil, cottonseed oil, castor oil, and lard [5, 6, 7, 8, 9 and 10].

In view of the above, it can be seen that no work is reported on the optimization of FFA content of JCO and its biodiesel yield using four process variables. The present paper, therefore, reports the results of the optimization of four process variables viz. catalyst (H_2SO_4) concentration (0-2% v/v), reaction temperature (40^o-60^{o}C), reaction time (30-180 min) and methanol/oil ratio (w/w) (3:1 – 12:1) for the esterification process of JCO containing high FFA (14.6%) and catalyst (NaOH) concentration (0-2% w/w), reaction temperature (35^o-55^{o}C), reaction time (30-180 min) and methanol/oil ratio (w/w) (6:1–12:1) for transesterification of JCO using RSM based CCD in 54 experimental runs with the help of Design Expert 8.0.6 software. A model to predict the response was formulated and validated by ANOVA. The model can be used in the industry to reduce the FFA content of JCO before carrying out its base catalyzed transesterification and improve the efficiency of biodiesel production, thereby, saving time and cost of the process in optimizing the process parameters.

II. Materials And Methods

JCO was procured from Jatropha Vikas Sansthaan, New Delhi. All chemicals like H_2SO_4, KOH, methanol, ethanol, Na_2SO_4, NaOH were of AR grade and 99% pure.

A. Esterification of JCO Containing High FFA Content

Raw JCO was filtered to remove all insoluble impurities followed by heating at 100°C for 10 min to remove all the moisture. The acid value of JCO was determined by the method used by Mahajan *et al*. [11]. The acid value was high i.e. 29.2 mg KOH g^{-1} corresponding to a FFA of 14.60%, which is far above the 1% limit for base catalyzed transesterification reaction. FFAs were, therefore, first converted to esters in a pretreatment process with methanol as solvent using conc. H_2SO_4 as acid catalyst by a process developed in the authors' laboratory for the production of JCB [12]. Hence, the high FFAs were first reduced to <1% using esterification and the resulting reaction mixture was subjected to base catalyzed transesterification process. In the present study, the esterification of JCO has been optimized using RSM for the reduction of FFAs.

B. Base Catalyzed Transesterification of JCO

Pretreated JCO (<1% FFA) was transesterified by using methanol as solvent and NaOH as base catalyst for the production of JCB [12]. The methyl ester layer was separated, washed with water, heated to remove moisture and dried over anhydrous Na_2SO_4. In the present study, the transesterification of JCO has been optimized using RSM for the production of JCB. The yield of JCB was calculated using the following equation (1):

$$\text{Yield of JCB (\%)} = \frac{\text{Total weight of methyl esters}}{\text{Total weight of oil in the sample}} * 100\% \quad (1)$$

C. Analysis of JCB

The JCB was prepared in the laboratory under the conditions optimized by RSM. The JCB was analyzed for fatty acid composition by Gas chromatograph (Model-Netal) using the process described by Jain and Sharma [12].

D. Physio-Chemical Properties of JCO and JCB

The physical and chemical properties of JCO and JCB produced under optimum conditions were determined by using standard methods [13].

E. Experimental Design

A five-level-four-factor CCD was applied for carrying out the optimization studies to reduce the FFAs of JCO in the esterification process and maximize the yield of JCB in the transesterification process respectively. A total of 54 experiments were conducted separately for getting the experimental response of FFA and JCB yield. The catalyst concentration (A) (%), reaction temperature (B) (°C), reaction time (C) (min) and methanol/oil ratio (w/w) (D) were the independent variables selected for optimization. The coded and uncoded levels of the independent variables used for the esterification and transesterification of JCO are given in Table I and Table II respectively.

TABLE I
INDEPENDENT VARIABLES USED FOR CCD IN ESTERIFICATION OF JCO

Variables	Symbols	Levels				
		-1	**-0.5**	**0**	**0.5**	**1**
Catalyst (H_2SO_4) concentration (% v/v)	A	0	0.5	1.0	1.5	2.0
Temperature (°C)	B	40	45	50	55	60
Time (min)	C	30	67.5	105	142.5	180
Methanol/oil ratio (w/w)	D	3	5.25	7.5	9.75	12

TABLE II
INDEPENDENT VARIABLES USED FOR CCD IN TRANSESTERIFICATION PROCESS

Variables	Symbols	Levels				
		-1	**-0.5**	**0**	**0.5**	**1**
Catalyst (NaOH) concentration (% w/w)	A	0	0.5	1.0	1.5	2.0
Temperature (°C)	B	35	40	45	50	55
Time (min)	C	30	67.5	105	142.5	180
Methanol/oil ratio (w/w)	D	6	7.5	9	10.5	12

F. Statistical Analysis

The Design Expert 8.0.6 software was used for the regression and graphical analysis of the data. The minimum values of FFA were taken as the response of the design experiment for esterification process and the maximum values of JCB yield were taken as the response of the design experiment for transesterification process. The experimental data obtained by the above procedure was analyzed by the response surface regression using the following second-order polynomial equation (2):

$$y = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i_{i>j}}^{k} \sum_{j}^{k} \beta_{ij} x_i x_j \quad (2)$$

where; y is the response, i and j are the linear and quadratic coefficients respectively, x_i and x_j are the uncoded independent variables, β_o is the regression coefficient, k is the number of factors studied and optimized in the experiment. Statistical analysis of the model was carried out to evaluate the analysis of variance (ANOVA). Equation was also validated by carrying out confirmatory experiments.

III. Results And Discussions

A. Esterification Process

The experimental and predicted values for FFA responses at the design points and all the four variables in uncoded form are given in Table I II. ANOVA results of the model showed that the associated Probability (P) value for the model was lower than 0.0001, thus, implying the significance of the model. The value of regression coefficient R^2 for the model was 0.96, indicating the good fitness of the model. The predicted R^2 was 0.92, further, proving the reliability of the model [14].

TABLE III

CCD ARRANGEMENT AND RESPONSES FOR ESTERIFICATION OF JCO

Run	A: Catalyst (H_2SO_4) concentration (%)	B: Temperature (^oC)	C: Time (min)	D: Methanol/oil ratio	Free Fatty Acid (%)	
					Experimental Response	Predicted Response
1	1	50	105	12	2.22	3.03
2	1.5	45	67.5	5.25	2.82	3.35
3	1.5	45	67.5	5.25	2.96	3.35
4	1.5	45	142.5	5.25	2.48	2.80
5	0.5	55	142.5	9.75	1.11	1.11
6	1	50	105	7.5	1.02	1.08
7	0.5	45	67.5	5.25	2.73	3.54
8	1.5	55	67.5	9.75	2.94	3.18
9	1	50	105	3	1.99	0.93
10	1.5	45	142.5	5.25	2.51	2.80
11	1.5	45	142.5	9.75	2.53	2.74
12	0.5	45	67.5	9.75	7.32	7.35
13	1	50	105	7.5	0.99	1.08
14	0.5	45	142.5	9.75	3.87	3.68
15	1	50	105	12	2.26	3.03
16	1	40	105	7.5	7.64	7.13
17	0.5	55	142.5	9.75	1.09	1.11
18	0.5	55	142.5	5.25	1.87	1.95
19	1	60	105	7.5	2.98	3.12
20	1.5	45	67.5	9.75	6.17	6.28
21	1.5	55	142.5	9.75	0.65	0.06
22	1	50	105	7.5	1.06	1.08
23	1	50	30	7.5	6.84	6.18
24	1	40	105	7.5	7.56	7.13
25	0	50	105	7.5	1.57	1.52
26	0.5	55	67.5	9.75	4.49	4.36
27	1	50	30	7.5	6.78	6.18
28	1.5	45	142.5	9.75	2.55	2.74
29	2	50	105	7.5	0.51	0.28
30	0.5	45	142.5	5.25	2.61	2.87
31	1	50	105	7.5	1.01	1.08
32	0.5	55	67.5	9.75	4.52	4.36
33	0.5	45	142.5	9.75	3.81	3.68
34	0.5	55	142.5	5.25	1.85	1.95
35	2	50	105	7.5	0.53	0.28
36	1	50	180	7.5	2.04	2.39
37	1.5	55	142.5	9.75	0.67	0.06

38	0.5	45	142.5	5.25	2.65	2.87
39	1	50	105	7.5	1.05	1.08
40	1	50	105	7.5	1.04	1.08
41	0.5	55	67.5	5.25	1.91	2.19
42	1.5	55	142.5	5.25	1.33	1.78
43	1	50	105	3	2.05	0.93
44	0.5	45	67.5	9.75	7.38	7.35
45	1.5	55	67.5	9.75	3.02	3.18
46	1	60	105	7.5	2.91	3.12
47	1.5	55	142.5	5.25	1.36	1.78
48	0.5	55	67.5	5.25	1.94	2.19
49	0.5	45	67.5	5.25	2.78	3.54
50	1.5	55	67.5	5.25	1.56	1.90
51	0	50	105	7.5	1.58	1.52
52	1	50	180	7.5	2.06	2.39
53	1.5	45	67.5	9.75	6.25	6.28
54	1.5	55	67.5	5.25	1.55	1.90

The regression equation (3) for the determination of predicted values of output parameter (i.e. FFA) is given as follows:

$$FFA\ (\%) = 102.64+1.55A-4.01B-0.11C+2.54D-0.01AB+0.002AC-0.20AD+0.001BC-0.037BD-0.009CD-0.18A^2+0.04B^2+0.006C^2+0.04D^2 \quad (3)$$

The graph between the predicted and actual FFA values given in Fig. 1, shows that the predicted values are quite close to the experimental values, thus, validating the credibility of the model developed for establishing a correlation between the process variables and the FFA content.

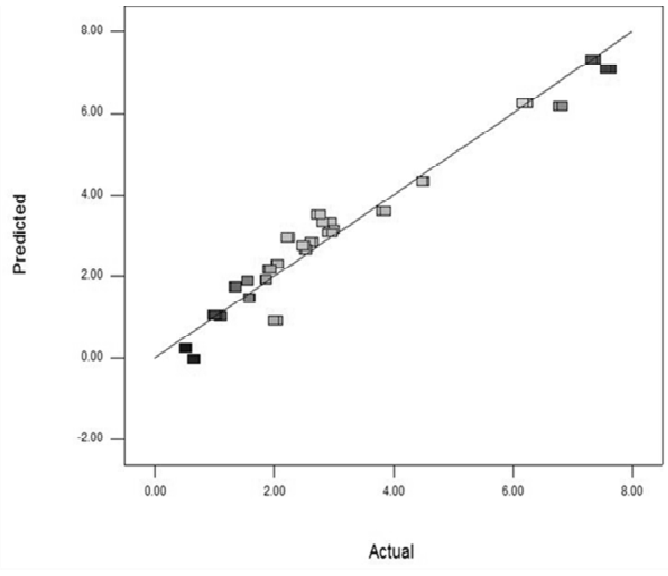

Fig. 1 Predicted Versus Actual FFA Values

1) Effect of Process Variables on FFA Content of JCO:

Fig. 2 shows the effect of catalyst (H_2SO_4) concentration, reaction temperature, time and methanol/oil ratio on FFA content. It can be seen from the figure that the FFA decreases with increase in catalyst concentration. FFA decreases with increase in temperature and time till the middle point is reached, beyond which it increases. Methanol/oil ratio is found to have very less effect on FFA content as the change in FFAs with change in the methanol/oil ratio is very small. So, very high temperature and long reaction time should be avoided as they have an inhibitive effect on the reduction of FFA content. These results were found similar to the work of Tiwari *et al*. [3].

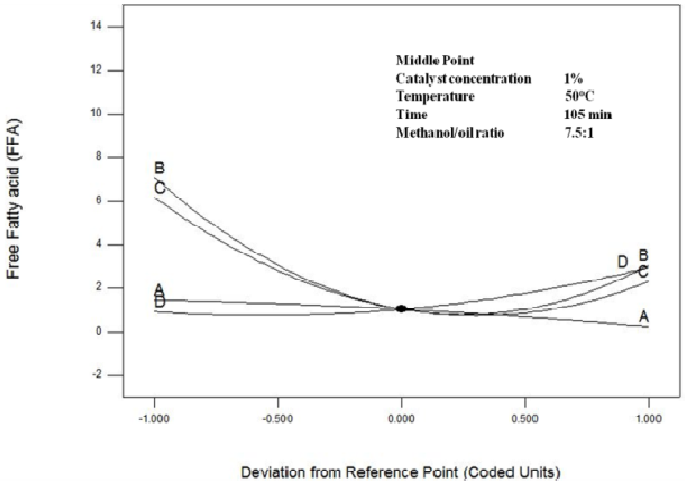

Fig. 2 Effect of Catalyst Concentration, Temperature, Time and Methanol/Oil Ratio on FFA (%) Content

B. Transesterification Process

Experimental and predicted values for JCB yield responses at the design points and all the four variables in

uncoded form are given in Table IV. The associated P value for the model was lower than 0.0001, thus, implying the significance of the model. The value of regression coefficient R^2 for the model was 0.945, indicating the good fitness of the model. A high value of predicted R^2 (0.890) is an indication of precision of fitted model. The more the value of R^2 approaches unity, the better the model fits the experimental data. [14].

TABLE IV

CCD ARRANGEMENT AND RESPONSES FOR TRANSESTERIFICATION OF JCO

Run	A: Catalyst (NaOH) concentration (%)	B: Temperature (°C)	C: Time (min)	D:Methanol/oil ratio	JCB yield (%)	
					Experimental Response	Predicted Response
1	1	45	105	9	82.6	82.84
2	0	45	105	9	89.5	89.81
3	0.5	40	67.5	10.5	78.2	80.37
4	1.5	40	142.5	10.5	88.6	87.84
5	0.5	40	67.5	10.5	77.8	80.37
6	0.5	50	67.5	7.5	87.1	86.76
7	1.5	50	67.5	10.5	73.9	76.50
8	0.5	40	142.5	7.5	91	89.83
9	1	45	105	9	82.9	82.84
10	0.5	40	67.5	7.5	86.1	87.33
11	1	45	105	9	83.1	82.84
12	1	45	105	12	85.5	84.97
13	1	45	105	9	82.1	82.84
14	1	45	180	9	93.4	92.65
15	0.5	50	142.5	10.5	97.8	99.99
16	1	45	30	9	78.2	79.56
17	1.5	50	142.5	10.5	88.3	87.10
18	0.5	50	67.5	10.5	90	87.17
19	0.5	40	142.5	10.5	91.8	91.86
20	1.5	40	67.5	7.5	85.7	84.18
21	0.5	50	142.5	7.5	90	90.98
22	1	35	105	9	92.4	91.00
23	1.5	40	142.5	7.5	81.6	84.05
24	1.5	40	142.5	10.5	83.8	87.84
25	1	45	105	12	85.9	84.97
26	0.5	50	67.5	10.5	90.4	87.17
27	1.5	40	67.5	10.5	80	78.97
28	2	45	105	9	73	73.37
29	1	55	105	9	88.2	89.69
30	1.5	50	142.5	7.5	77	75.94
31	1.5	40	67.5	7.5	85.2	84.18
32	1	45	105	9	82.4	82.84
33	1	55	105	9	87.9	89.69
34	1.5	50	67.5	10.5	74.1	76.50
35	1.5	50	67.5	7.5	74.8	74.34
36	1.5	50	142.5	7.5	77.3	75.94

37	1.5	40	67.5	10.5	80.2	78.97
38	1	45	105	9	82.8	82.84
39	0.5	50	67.5	7.5	87.5	86.76
40	2	45	105	9	73.2	73.37
41	1	45	105	6	79.5	80.76
42	1.5	50	67.5	7.5	74.5	74.34
43	0	45	105	9	89.9	89.81
44	1.5	40	142.5	7.5	81.2	84.05
45	0.5	50	142.5	7.5	90.4	90.98
46	0.5	50	142.5	10.5	98.2	99.98
47	1	35	105	9	92.1	91.00
48	1	45	105	6	79.8	80.76
49	0.5	40	67.5	7.5	86.5	87.33
50	0.5	40	142.5	10.5	92.1	91.86
51	1	45	30	9	78.5	79.56
52	0.5	40	142.5	7.5	91.4	89.83
53	1.5	50	142.5	10.5	88.7	87.10
54	1	45	180	9	93.7	92.65

The regression equation (4) for the determination of predicted values of output parameter (i.e. JCB yield) is given as follows:

$$JCB\ (\%) = 343.90+34.43A-8.34B-0.46C-15.19D-0.93AB-0.035AC+0.58AD+0.0023BC+0.25BD+0.04CD-1.25A^2+0.075B^2+0.0006C^2+0.0023D^2 \quad (4)$$

The graph between the predicted and actual JCB yield (%) given in Fig. 3 shows that the predicted values are quite close to the experimental values, thereby, validating the reliability of the model developed for establishing a correlation between the process variables and the JCB yield.

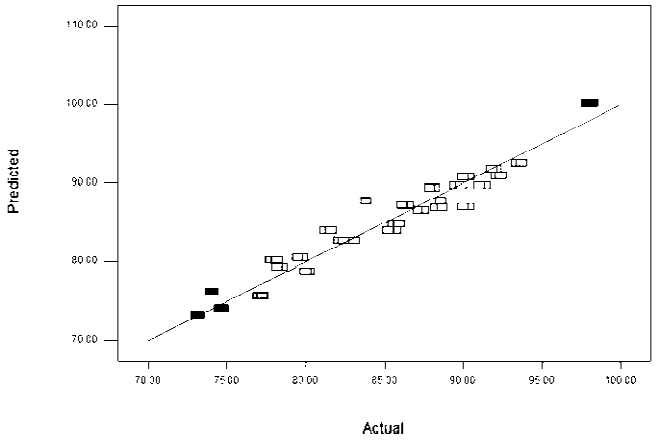

Fig. 3 Predicted Versus Actual JCB (%) Yield Values

1) Effect of Process Variables on JCB Yield:

Fig. 4 shows the effect of catalyst concentration (NaOH), reaction temperature, reaction time and methanol/oil ratio on JCB yield. It can be seen from the figure that the JCB yield decreases significantly with increase in catalyst concentration. This may be due to the fact that addition of excessive catalyst favors the saponification reaction of triglycerides to form soap which decreases the biodiesel yield [15]. JCB yield decreases with increase in temperature initially and increases at higher values of temperature. The increase in biodiesel yield at higher temperature is due to the fact that viscosity of oils decreases at high temperature which results in an increased reaction rate, thus, increasing the biodiesel yield [15]. JCB yield increases with increase in time. This can be supported by the work of Freedman *et al*. [16] who found that the conversion rate of fatty acid esters increases with reaction time. JCB yield is found to increase with the increase in methanol/oil ratio; since the transesterification reaction is reversible in nature, so excess alcohol is added to ensure the total conversion of triglycerides [15]. Thus, the yield of biodiesel increases with increase in methanol quantity.

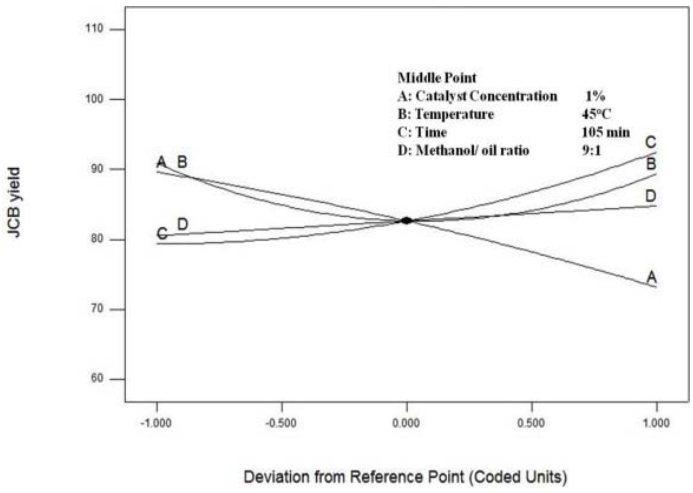

Fig. 4 Effect of Catalyst Concentration, Temperature, Time and Methanol/Oil Ratio on JCB (%) Yield

C. Optimization of Response Parameters

Optimization of individual responses was performed separately to achieve the desired reduction in FFA content and maximization of JCB yield based on the respective developed mathematical equations. The optimal value of input process parameters is given in Table V. Predicted response is found to be in good agreement with the experimental results.

TABLE V

OPTIMIZED INPUT PROCESS PARAMETERS AND OPTIMUM VALUE OF JCB YIELD

Response	Optimized value of input process parameter				Predicted value	Experimental value
	A	B	C	D		
FFA (%)	1.5	50	125	6.5	0.37	0.34
JCB yield (%)	1.0	55	110	11	98.89	98.3

D. Analysis of JCB

The Fatty acid (FA) composition of JCB prepared under the above optimum parameters and determined by Gas Chromatography (GC) is given in Table VI which shows that JCB mainly contained Oleic and Linoleic acid. The FA composition is in good agreement with the composition reported by Jain and Sharma [13]. The physio-chemical properties of JCO and JCB are reported in Table VII. The properties of JCB are in good agreement with ASTM and IS specifications.

TABLE VI

FATTY ACID COMPOSITION OF JCB

Fatty acid	Formula	% Composition
Palmitic acid	$C_{16}H_{32}O_2$ $CH_3(CH_2)_{14}COOH$	16.2
Stearic acid	$C_{18}H_{36}O_2$ $CH_3(CH_2)_{16}COOH$	8.2
Oleic acid	$C_{18}H_{34}O_2$ $CH_3(CH_2)_7$-CH=CH-$(CH_2)_7COOH$	38.4
Linoleic acid	$C_{18}H_{32}O_2$ $CH_3(CH_2)_4CH$=CH-CH_2-CH=CH-$(CH_2)_7COOH$	36.8
Linolenic acid	$C_{18}H_{30}O_2$ $CH_3(CH_2)_4CH$=CH-CH_2-CH=CH-CH_2-CH=CH-$(CH_2)_4COOH$	0.4

TABLE VII

PHYSIO-CHEMICAL PROPERTIES OF JCB

S.no	Property (unit)	ASTM D6751	IS 15607	JCO	JCB prepared under optimum parameters	JCB [9]	ASTM D6751 limits	IS 15607 limits
1	Viscosity (cSt; 40 °C)	ASTM D445	IS 1448	50	4.9	4.38	1.9-6.0	2.5-6.0
2	Density (g/c.c at 15°C)	ASTM D4052	IS 1448	0.930	0.862	-	-	0.860-0.900
3	Flash point (oC)	ASTM D93	IS 1448	241	174	172	Min. of 130	-
4	Water and Sediment (Vol%)	D2709	D2709	-	0.05	0.05	Max. of 0.05	Max. of 0.05
5	Free glycerin (% mass)	D6584	D6584	-	0.01	0.01	Max. of 0.02	D6584
6	Oxidative stability of JCB (h)	EN14112	-	-	3.3	3.27	3	-
7	Free glycerol	D6584	D6584	--	0.015	0.01	Max. of 0.02	Max. of 0.02
8	Total glycerol	D6584	D6584	-	0.14	0.12	Max. of 0.25	Max.of 0.25
9	Ester content (%)	-	EN 14103	-	98.3	98.5	-	Min. of 96.5

IV. CONCLUSIONS

Optimization studies for reduction of FFA of JCO and maximization of yield of JCB (%) were carried out. Process optimization was accomplished by five level-four-factorial CCD using RSM. The high FFA (14.6%) of JCO was reduced to 0.34% by its pretreatment with methanol (6.5:1) using H_2SO_4 as catalyst (1.5% v/v) in 125 min time at 50^{o}C temperature. A biodiesel yield of 98.3% was achieved with methanol/oil molar ratio (11:1) using NaOH as catalyst (1% w/w) in 110 min time at 55^{o}C temperature. Effects of catalyst concentration, reaction temperature, reaction time and methanol/oil ratio were studied on the esterification and transesterification processes. Second-order model equations

were obtained to predict the FFA content and JCB yield as a function of input parameters. On the basis of ANOVA; the catalyst concentration, reaction time and methanol/oil molar ratio had a significant effect on JCB yield. The models can be successfully employed in the oil industry to reduce the FFA content of JCO before carrying out base catalyzed transesterification, thereby, saving time and maximize the yield of methyl esters respectively. The prepared JCB conformed to the ASTM and IS standards specifications.

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